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PECTIN
CHEMISTRY, FUNCTIONALITY, & APPLICATIONS

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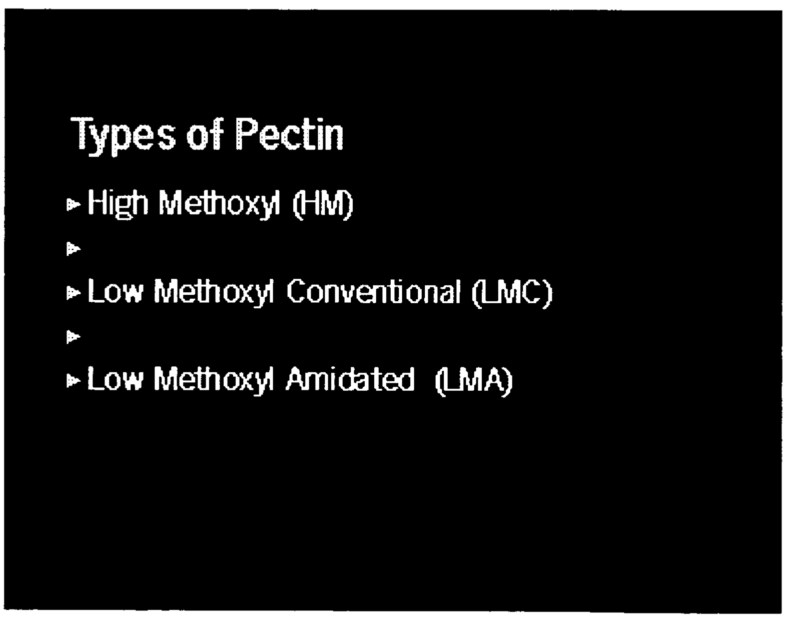
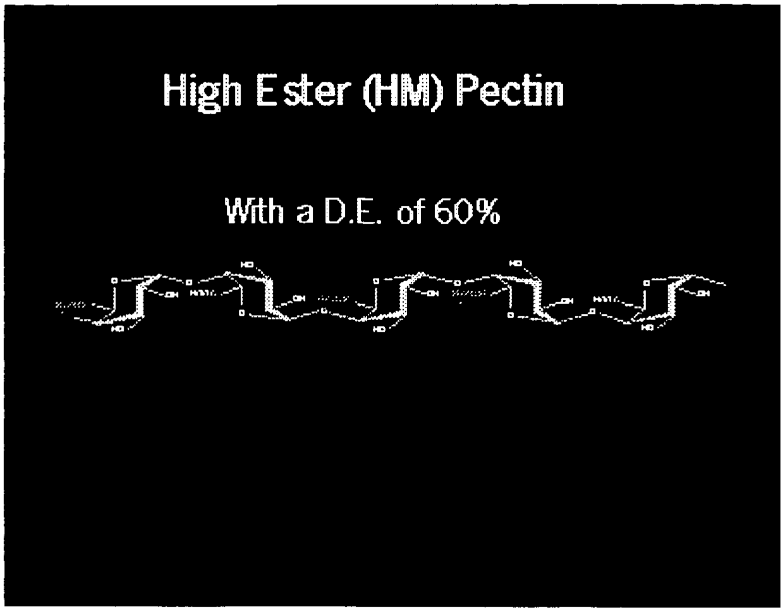
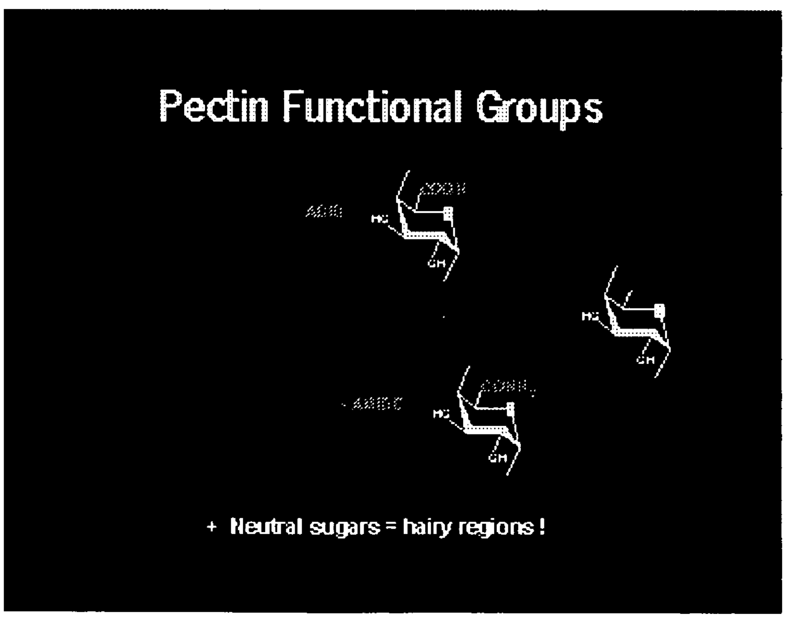
In this lecture all types of pectin will be discussed, however the emphasis will be on LM pectin, as this is the most misunderstood yet most versatile type of pectin. Applications of both types of pectin in food systems will be covered at the end of the talk.



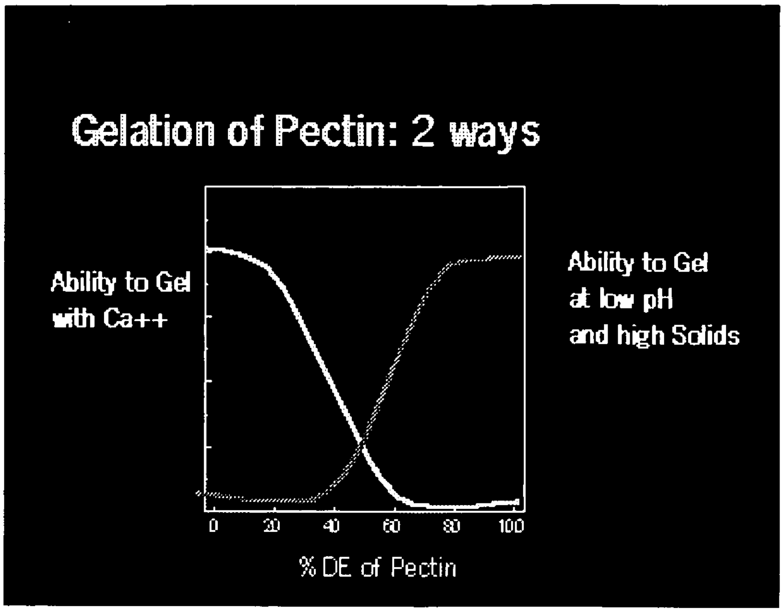
Pectin is divided into two main categories: HM pectin and LM pectin (Figure 1). The LM pectins are further subdivided into two groups: low methoxyl amidated (LMA), and low methoxyl conventional (LMC). The reasons for these three classes of pectin will become clear as we get into the chemistry of pectin. Some biochemistry will be covered at the beginning of the talk, but no more than is necessary for your understanding of why pectin behaves the way it does.



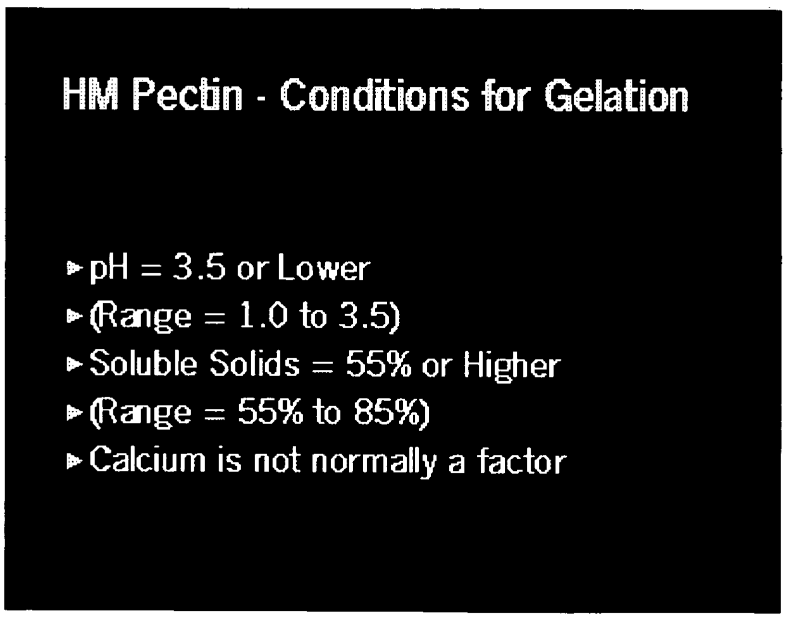
Pectin is the methylated ester of polygalacturonic acid. It is commercially extracted from citrus peels and apple pomace under mildly acidic conditions. Figure 2 shows a portion of a pectin molecule. Each ring is a molecule of galacturonic acid, and there are 300 to 1000 such rings in a typical pectin molecule, connected in a linear chain. You can see five such galacturonic acid units in Figure 2. Please note that three of the five are in the methyl ester form, while the other two are in the acid form. This represents a degree of methoxylation of 3 out of 5, or 60 percent. You will see the term abbreviated as "DM" or "DE", which is short for degree of esterification. Both terms are interchangeable, and they refer to the percentage of acid groups which are present in the pectin molecule as the methyl ester.



The "business end" of the pectin molecule is it's carboxyl acid group. As seen in Figure 3, the only difference between HM pectin and LMC pectin is in the relative proportions of acid and ester groups, yet it is this difference that causes them to gel under completely different conditions. The LMA pectins may have up to 25% amide groups, and this changes their texture and temperature characteristics, which will be explained a little further on.



By FCC definition, any pectin of 50% DE or greater is a High Methoxyl pectin, while anything under a DE of 50% is low methoxyl pectin. The two types of pectin will gel for completely different reasons, as indicated in Figure 4. HM pectin gels due to high soluble solids and low pH conditions, as indicated on the graph as a solid line. As the DE of a pectin is lowered, it begins to lose it's ability to gel under these conditions. The dotted line is for the ability to gel with divalent ions (usually calcium ions in food systems). This is the hallmark of LM pectin. Please note that as the DE is raised, the pectin will eventually lose it's ability to gel with calcium. Also note that a pectin with a DE around 50% will posses characteristics of both



The bare minimum conditions for causing HM pectin to gel are shown in Figure 5. If your system is not at least 55% solids AND has a pH of 3.5 or lower, then HM pectin will not gel, no matter how much of it you add to your product. I'm not saying that HM pectin isn't used under these conditions, but if it is, then it is not a gelling agent but a thickening agent. From Figure 5 we can see that the range of gelling conditions for HM pectin are a pH of 1.0 to 3.5, and a solids range of 55% to 85%. Also note that the presence or absence of calcium ions is not normally a factor for HM pectin gelation, except in special cases.

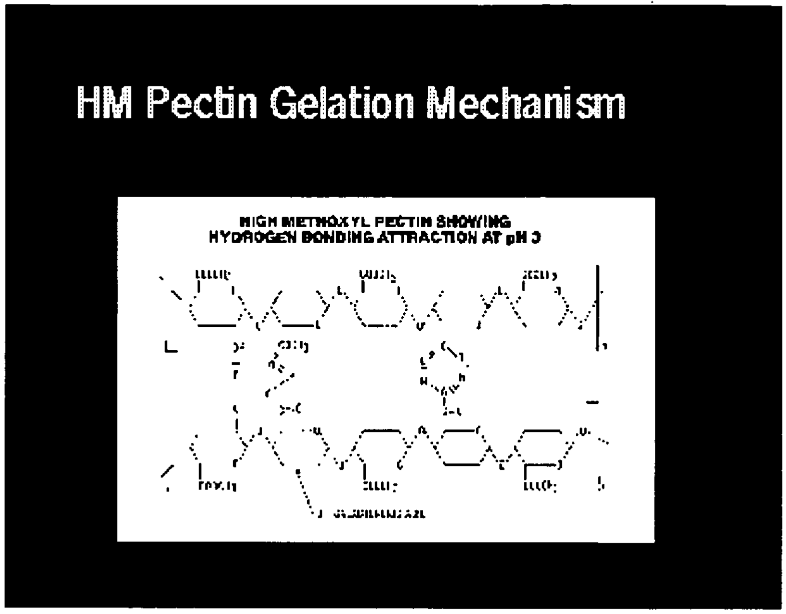
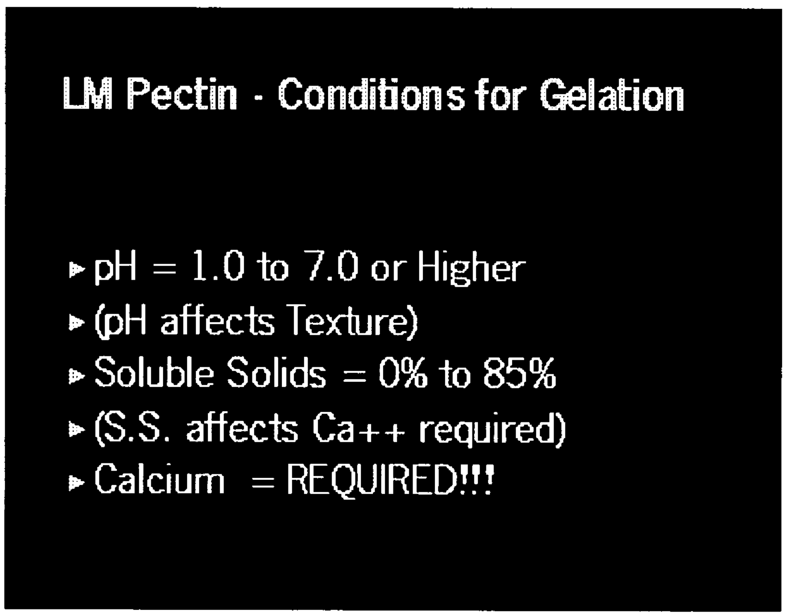


Figure 6 illustrates the mechanism of gelation for HM pectin. At a pH of 3.0, about 90% of the available acid groups are not dissociated, and are therefore capable of hydrogen bonding to acid or hydroxyl groups on adjacent chains. These "junction zones" could be thought of as crystallized out of solution, while the non-cross linked portions of the molecules are still in solution. Therefore, it could be said that an HM pectin gel is literally half in and half out of solution.



The gelling range of conditions for LM pectin is illustrated in Figure 7. The good news is that LM pectin gels over a wider pH range than HM pectin, namely pH = 1.0 to 7.0 or higher. pH does influence the texture of the gel, which I will explain later. Also, the soluble solids or Brix gelling range for LM pectin is much wider than HM pectin. One can gel LM pectin from 0% to 80% solids. The bad news is that you have one more parameter to keep track of, and that is the calcium content of your product. "No calcium, no gel" with LM pectin. Fortunately, this is not as difficult as it sounds, and this will be made clear shortly.

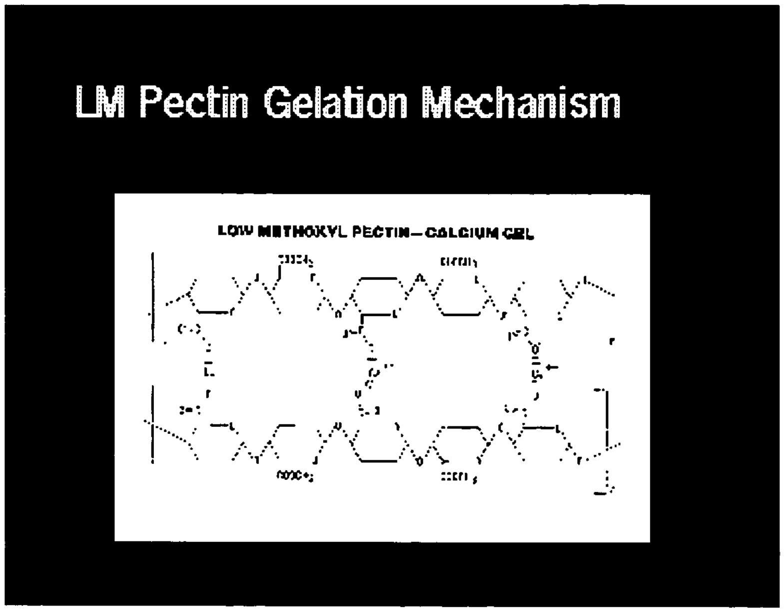


Figure 8 shows the gel mechanism for LM pectin. This involves joining carboxyl groups on adjacent chains with divalent ions, usually calcium or magnesium. Again, this creates a "junction zone" which can be thought of as crystallized out of solution. The "egg box" model for calcium alginate gels would also be valid for LM pectin gels.

LM Pectin - Calcium Reactivity

Type	D.E.	D.F.A.	Ca++ Reactivity	Ca++ required at 30% S.S.
LM12CG	31+-4	69+-4	High	25 mg/gm
LM18CG	40+-4	60+-4	Medium	50 mg/gm
LM22CG	49+-4	51+-4	Low	100 mg/gm

Figure 9 lists some parameters for 3 types of LMC pectin. These three pectins are a homologous series, differing only in their degree of methoxylation. The "DFA" is the degree of free acid, or percent carboxyl groups available for cross linking with calcium. By definition, the LM12CG pectin is said to have **HIGH** calcium reactivity, meaning that it needs **LESS** calcium than the other two LMC pectins to make a good gel, all other factors being constant. This makes sense because LM12CG has more acid groups along it's chain, therefore the statistical odds of a calcium ion being in the right place at the right time are greater. LM22CG has only 50% acid groups along it's chain, therefore the statistical odds for a calcium ion to be in the right place at the right time are lower. To raise the statistical odds, one must add more calcium to the LM22CG system, thus LM22CG needs more calcium than LM12CG, and is said to be **LESS** calcium reactive. To demonstrate the magnitude of the difference in calcium requirement, look at the last column of data. At 30% soluble solids, an LM12CG containing gel will need about 25 mg of calcium ions for every gram of pectin. At 30% solids, the LM18CG gel will need about twice as much calcium, and the LM22CG gel will need three or four times as much, to make the same firmness of gel as the LM12CG with 25 mg per gram. Needless to say, only the LM12CG is actually intended for use at 30% solids.

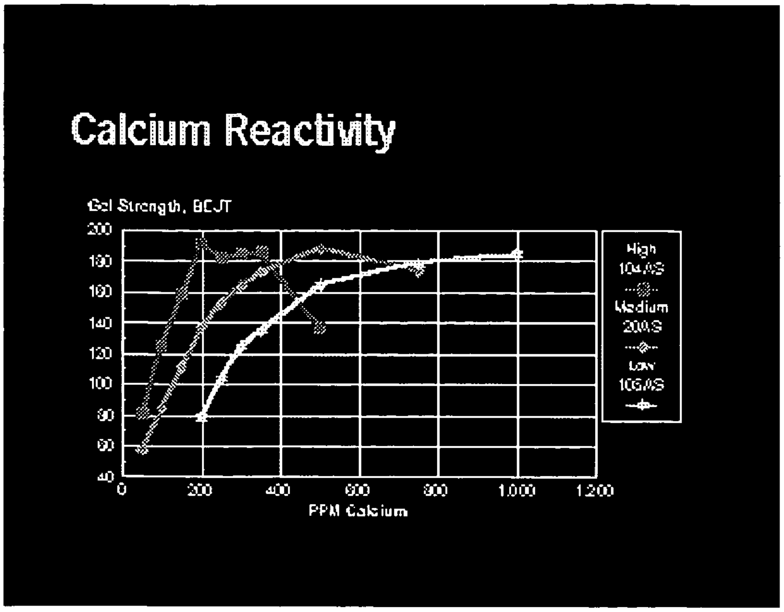


Figure 10 visually illustrates the relationship between LM104AS, LM20AS, and LM105AS at 30% solids. Note that the calcium response curves of each pectin "flatten out" at some point. This is known as **calcium saturation**, where an additional increment of calcium has no additional effect on the strength of the LM pectin gel. Unlike other calcium gelling hydrocolloids such as sodium alginate, this saturation point is typical for all types of LM pectin.

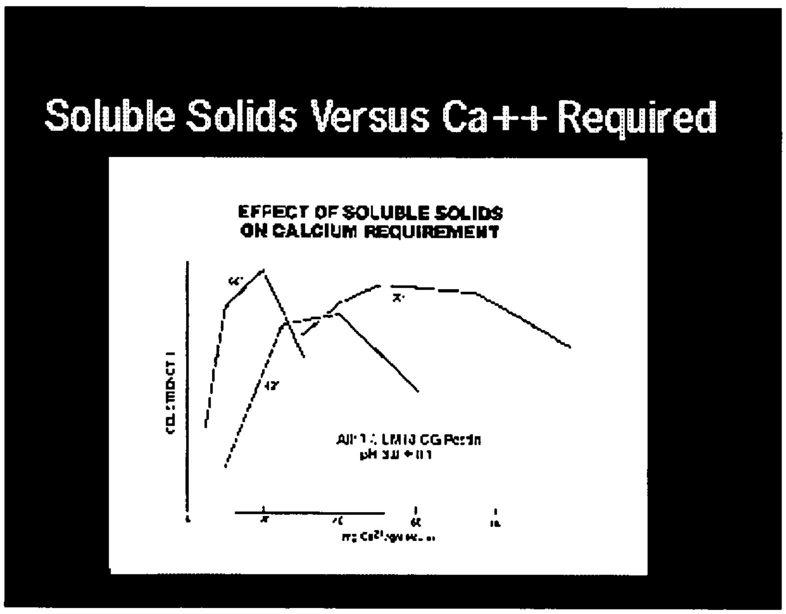


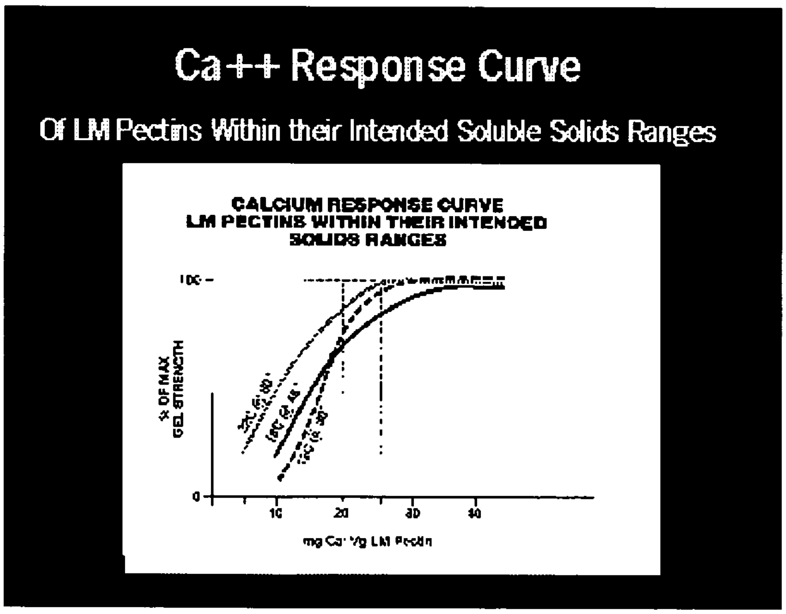
Figure 11 illustrates the effect of soluble solids on the amount of calcium required to make a proper gel. In our lab, when we want to test an LM pectin for it's response to calcium, we prepare five or six small batches of gel. Each batch contains the same amount of pectin, water, sugar, and buffer. The only difference between the six gels is the amount of calcium added. After 18 hours, we measure the firmness of each batch, and plot the data as gel strength versus calcium level. From Figure 11, one can see that at 30% solids with LM18CG pectin, about 40 to 100 mg calcium per gram of pectin are required to make a good gel. If we prepare the gels at 45% solids, the required calcium range drops down to about 20 to 45 mg calcium per gram pectin. At 60% solids, the requirement drops further to about 5 to 20 mg calcium per gram. With a given LM pectin, as the soluble solids goes up, the calcium requirement goes down. Please also note that as the soluble solids goes up, the calcium "bandwidth", or the "usable working range" of the pectin becomes more narrow. This can limit you from using a high calcium reactivity LM pectin at high solids levels: the calcium "bandwidth" becomes too narrow, and you can't keep your product within the required calcium range. Note that the "down" side of the calcium curve represents pregel, an

applesauce like texture which one usually tries to avoid. Pregel will be explained in detail later.

Suggested Pectin Types at Various Soluble Solids

Solids Range	LMC Type	LMA Type
0 to 45%	LM12CG	LM104AS
45 to 70%	LM18CG	LM20AS
55 to 85%	LM22CG	LM101AS

As a general rule, we recommend high calcium reactivity LM pectins for use at low soluble solids, and low reactivity LM pectins for higher soluble solids ranges. Figure 12 lists the typical LMA and LMC pectins, and their intended solids ranges.



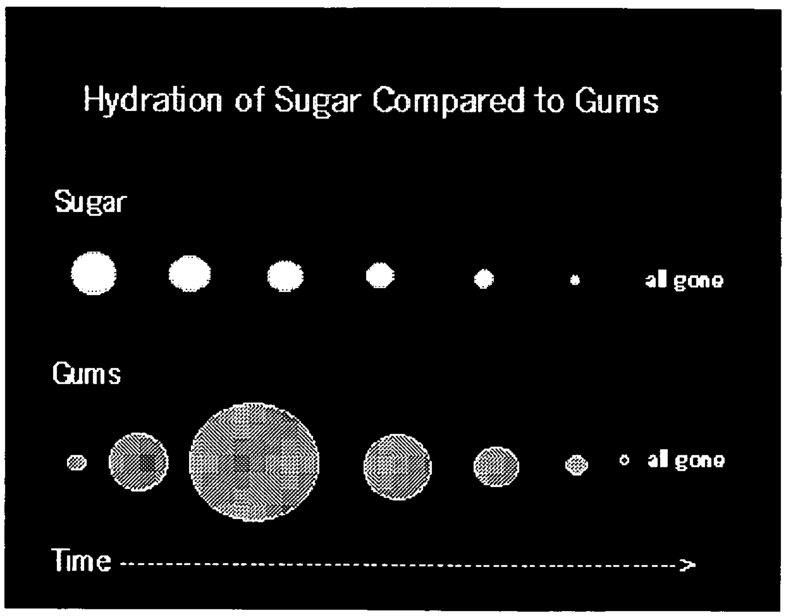
The idea of having several grades of LM pectin, which all perform the same functions, is so the end user only has to remember one calcium requirement. This is illustrated visually in Figure 13. The calcium response curve for LM12CG at 30% solids is the same as the response curve for LM18CG at 45% solids, which in turn is the same for the LM22CG response curve at 60% solids. We pair up the calcium reactivity with the soluble solids, so that the end user only has to remember one calcium response curve: that about 20 to 25 mg of calcium ions are needed for every gram of LM pectin present in the formula, to ensure efficient use of the LM pectin.

Making Pectin Solutions

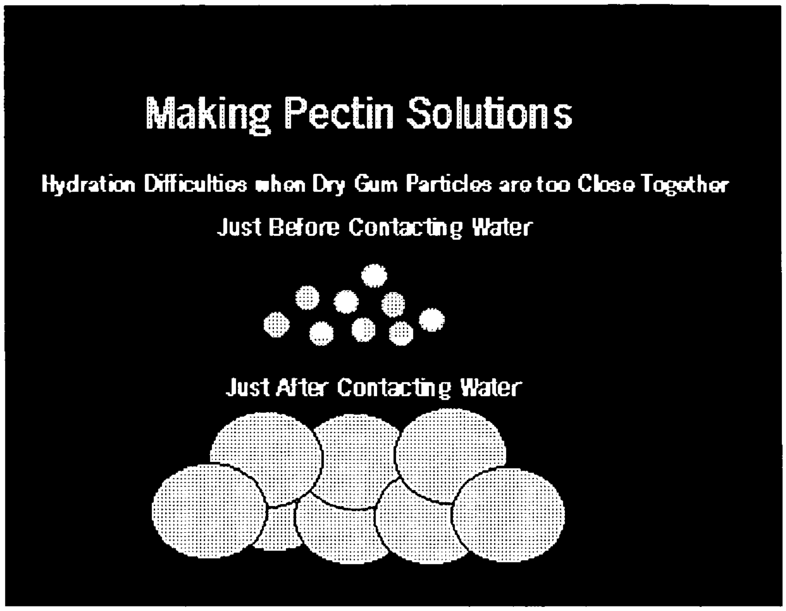
The idea is to slightly separate the particles from each other JUST BEFORE they hit the water.

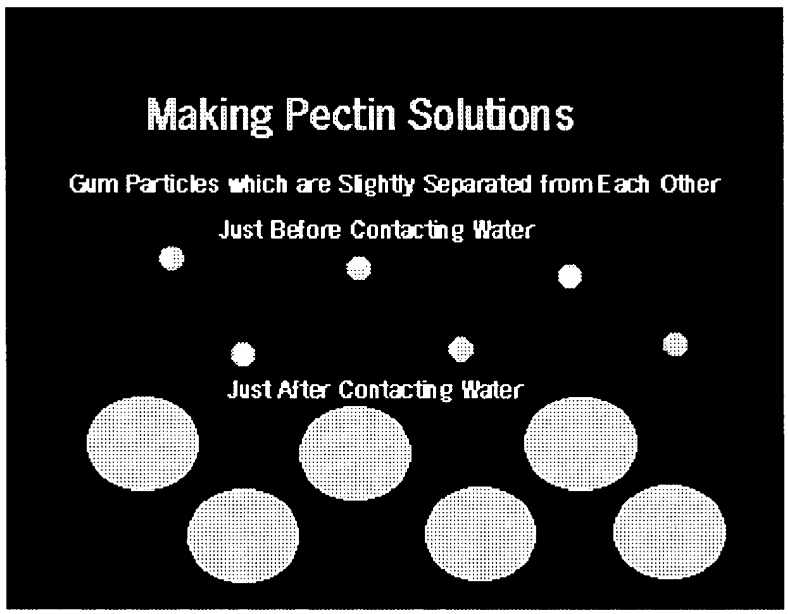
Method	Maximum % Pectin in Water
High Shear	10%
Hercules Eductor	7%
5/1 Sugar/Pectin Dry Blend	5%
Non-Solvent	5%

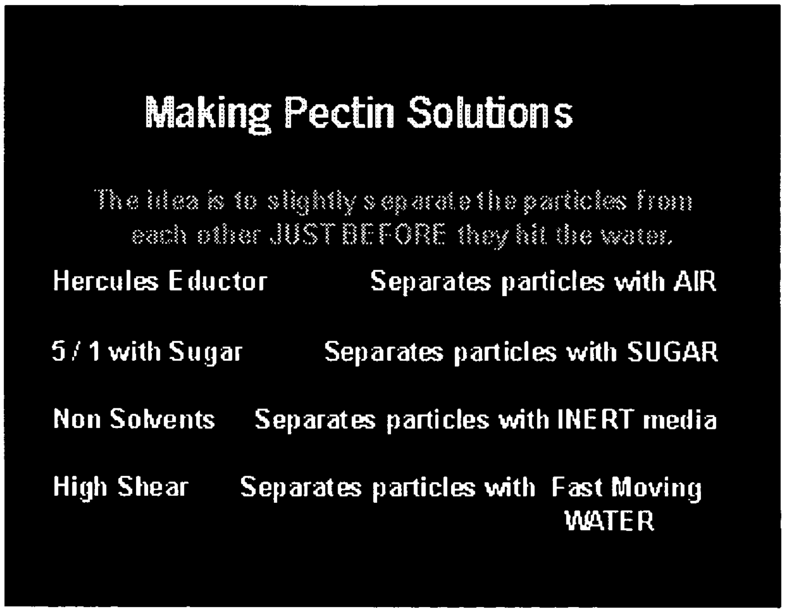
We will now take a break from LM pectin technology to review the proper means of hydrating pectin. If you try to stir a teaspoon of pectin into a beaker of water, you will get one large, sticky lump floating around in your beaker. If you are patient, and are willing to wait several days, the lump will eventually dissolve and go completely into solution. Most of us don't have the luxury of that much time to dissolve our pectin. This difficulty in dispersion holds true, not just for pectin, but for all hydrocolloids.



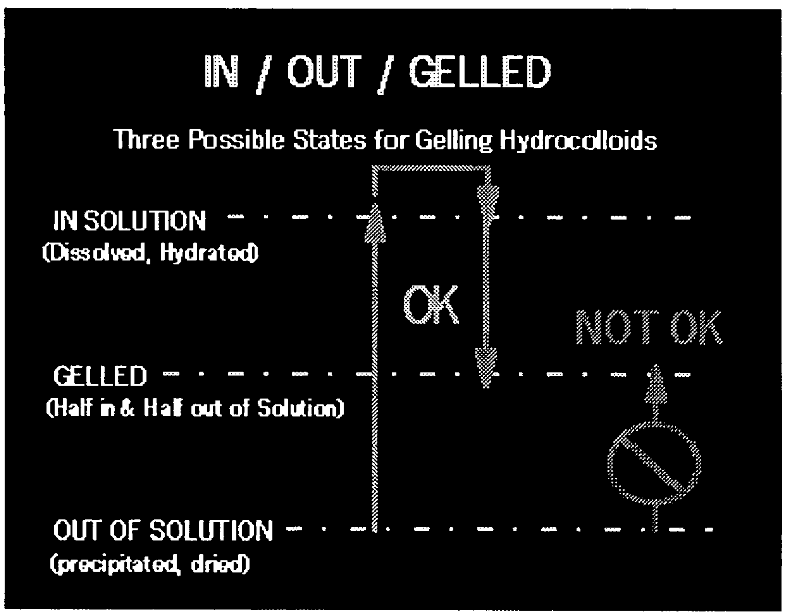
The key to lump-free pectin hydration is to remember the following: Separate the pectin particles from each other **BEFORE** they hit the surface of the water. Figure 15 shows a comparison of the hydration of the sugar in your morning coffee versus pectin. A sugar particle enters the water, and begins to dissolve from the outside in. The sugar particle becomes smaller with time as the molecules hydrate and float away, and within minutes all the sugar is dissolved. Pectin and other gums **DO NOT WORK THIS WAY!!!** When a pectin particle lifts the water, it rapidly absorbs water like a sponge and the particle swells to many times it's original size. I think of it as going ***"SPROINK"*** as it hits the water, becoming hundreds of times larger. When it has swelled to a certain size, then the pectin molecules begin to unravel themselves from the outside surface, and float away from the particle, being now completely hydrated. If the pectin particles are right next to each other when they contact the water, then they all try to ***"SPROINK"*** at the same time, and weld themselves together into one large, slow to hydrate lump. If the pectin particles are all slightly separated from each other when they contact the water, then they all have enough room to go through their initial expansion without getting stuck to a neighbor.


Making Pectin Solutions
Hydration Difficulties when Dry Gum Particles are too Close Together
Just Before Contacting Water
Just After Contacting Water

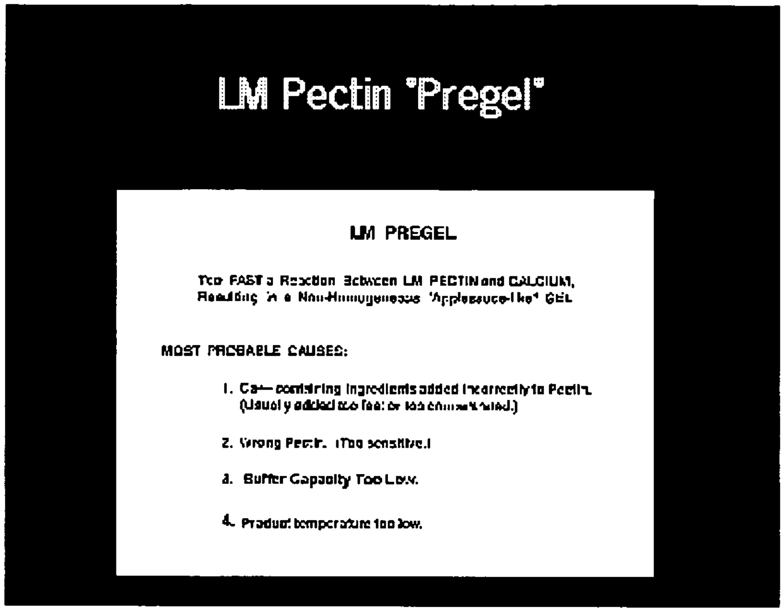

Making Pectin Solutions
Gum Particles which are Slightly Separated from Each Other
Just Before Contacting Water
Just After Contacting Water



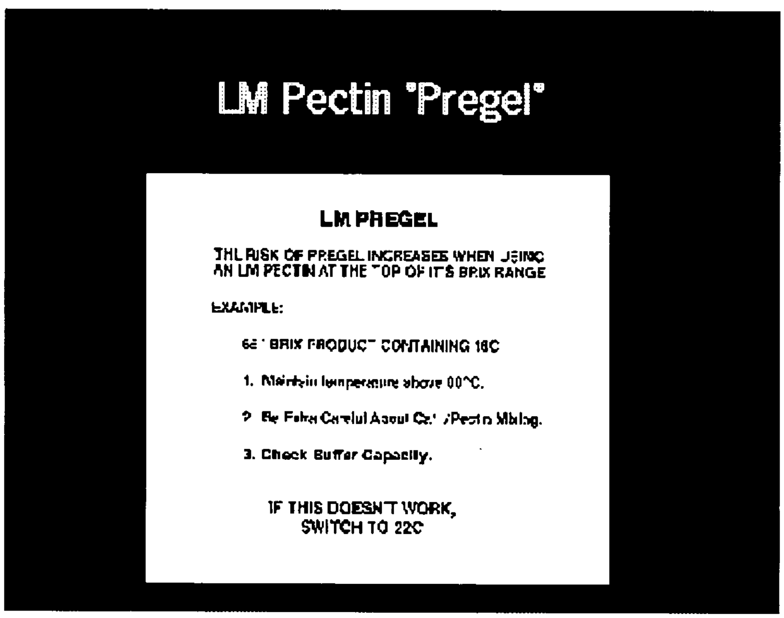
There are several ways to achieve this slight separation (Figure 16). The first is the use of a polymer disperser, such as the Hercules Eductor Funnel. Here, the pectin particles are separated by a stream of air just before they contact the water. Second is the dry blending of 5 parts sugar to 1 part pectin. When this is dispersed into water, the sugar particles (which don't go "***SPROINK***") separate the pectin particles, allowing the pectin to expand without contacting a pectin neighbor. Third is the use of non-solvents, such as vegetable oil, glycerin, or 80% solids 42DE corn syrup. With non-solvents, the pectin particles are wetted and separated from each other but cannot swell. Fourth is the use of high shear, where the rapidly moving water separates the gum particles. Also, if lumps were to form, the high level of mechanical work being done will break up lumps and ensure quick hydration. This method is typified by devices such as the Warring Blender, the Cuisinart food processor, the Breddo Likwifier, and the Clover Triblender.



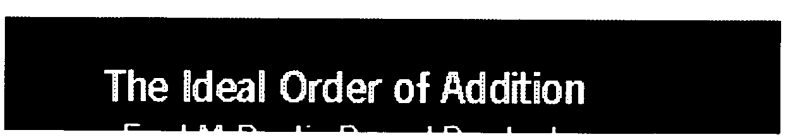
When you buy a drum of pectin, it is in the dried or precipitated state (Figure 17). The first thing you must do is to put it completely into the dissolved (hydrated) state, before you can induce it to go into the gelled state, which I think of as being "half in and half out" of solution. Mother Nature will not allow you to go from the dried state directly into the gelled state. To put it another way, you **CANNOT** hydrate a gelling agent under gelling conditions. You must hydrate it **OUTSIDE** of gelling conditions, and then induce gelling conditions by lowering the temperature, or adding calcium, or whatever it takes to trigger your particular gelling agent.



LM pectin pregel is defined as too fast a reaction between the LM pectin and the calcium ions, resulting in a nonhomogeneous gel structure, which resembles applesauce in texture (Figure 18). Nine times out of ten the cause of pregel is related to the way in which the calcium is introduced to the LM pectin. Usually, the calcium gets added too fast, or at the wrong point in the process, or it's simply too concentrated when added. About one time in ten, the LM pectin is too calcium reactive for the particular application, and a less reactive grade is required. Also, certain applications such as "conserves" are prone to pregel if their buffer capacity is too low. Very rarely, the cause is too low a process temperature.



For example, if you were to use LM18CG pectin (Figure 19) in a product at 65% soluble solids, and if you encountered pregel, then you would check the following: First, make sure your process temperature was 80 degrees C or higher. Next, make sure that you are using the proper order of addition of ingredients, which I will illustrate shortly. If these are OK, then the solution is to switch to LM22CG, which is the next less calcium reactive LM pectin in the series. Also, I would check the buffer capacity of my product, and if it was below 1.2%, I would add sufficient citric acid and sodium citrate to bring it to at least 1.2% without changing the final pH of the product.



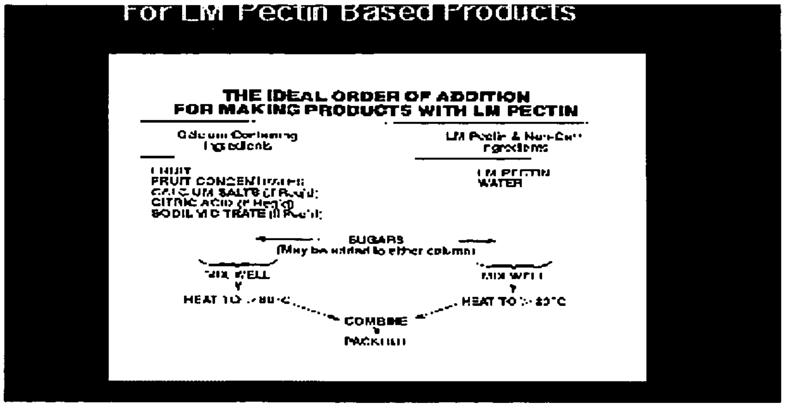
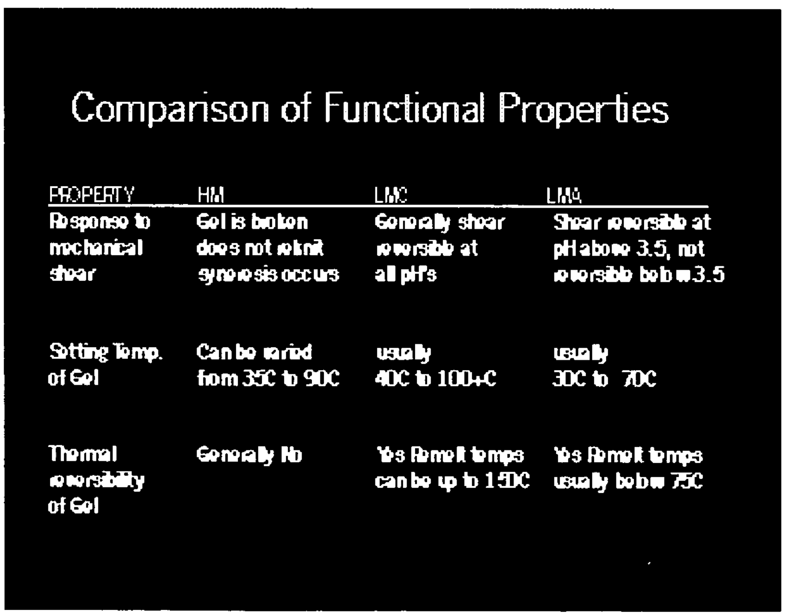


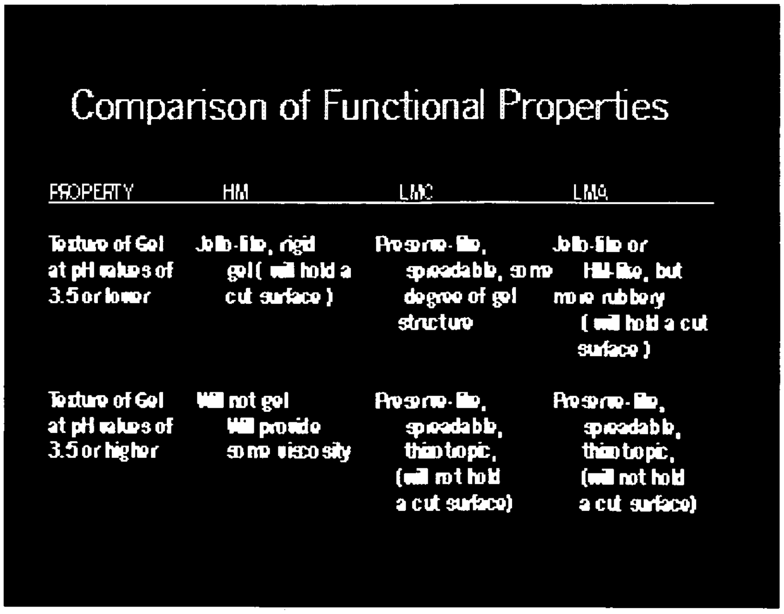
Figure 20 shows the ideal order of addition for LM pectin based products. It essentially comes down to this: Put all the calcium containing ingredients and all buffers in one pot on the stovetop, mix well, and heat to about 70 or 80 degrees C. Put water and the LM pectin into a Warring blender, and make a pectin solution. Put the solution into a second pot on the stovetop, and also heat to 70 or 80 C. When both pots are up to temperature, pour one into the other and gently mix. The idea here is to dilute out the calcium in as much other material as possible, to slow down the reaction rate. The pectin is "diluted" (actually it's hydrated) in water as much as possible, and the two fractions are heated to try to bring them above the gelling temperature of the system before they are combined. Since sugar contains virtually no calcium, it may be added to either pot or both pots, whatever is more convenient. Usually sugar is added to the fruit side, so as to osmotically equilibrate the system. That's all there is to it. There are some exceptions to this order of addition, but not many.


Comparison of Functional Properties

PROPERTY	HM	LMC	LMA
Response to mechanical shear	Gel is broken does not reknit syneresis occurs	Generally shear reversible at all pHs	Shear reversible at pH above 3.5, not reversible below 3.5
Setting Temp. of Gel	Can be varied from 35C to 90C	usually 40C to 100+C	usually 30C to 70C
Thermal reversibility of Gel	Generally No	Yes Remelt temps can be up to 150C	Yes Remelt temps usually below 75C

Figures 21 and 22 summarize the similarities and differences between the three major types of pectin. HM pectin gels are not shear reversible. If you stir a jar of jelly with a spoon, you will break the gel, and it will not re-knit over time. Instead, it will synerese over time. LMC pectin is generally regarded as shear reversible over the whole pH range, while LMA pectin is shear reversible at pH values of 3.4 or higher.

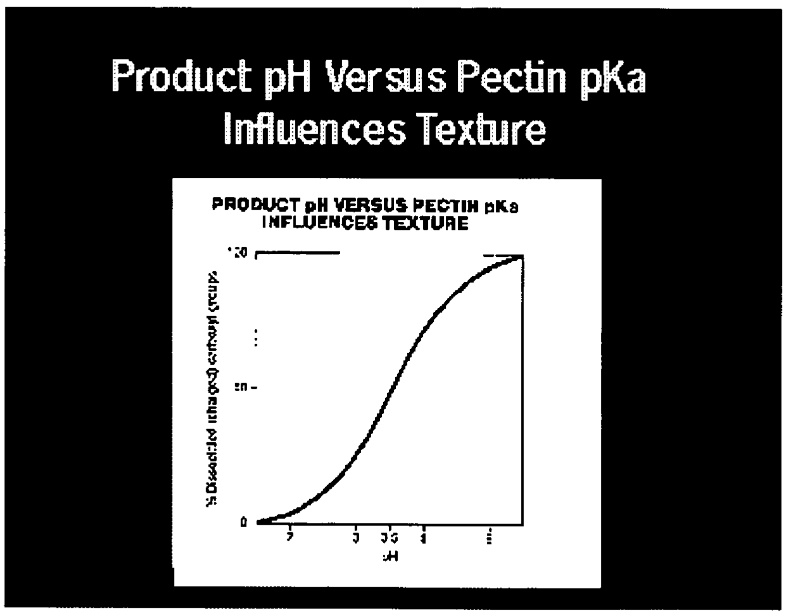
The setting temperature of an HM pectin gel can be varied between the limits of 25C to 90C, by changing the DE of the pectin. An LMC gel will usually set between the limits of 40C to 100+C, while an LMA gel will generally set between 30C to 70C. As far as thermal reversibility is concerned, HM gels are not thermally reversible (i.e., they don't melt when reheated). LMC and LMA gels are thermally reversible, and LMA gels generally re-melt by the time they have been reheated to 75C. LMC gels can have re-melt temperatures of up to 150C, so they can appear to be thermally irreversible under atmospheric conditions.



Comparison of Functional Properties

PROPERTY	HM	LMC	LMA
Texture of Gel at pH values of 3.5 or lower	Jello-like, rigid gel (will hold a cut surface)	Preserve-like, spreadable, some degree of gel structure	Jello-like or HM-like, but more rubbery (will hold a cut surface)
Texture of Gel at pH values of 3.5 or higher	Will not gel. Will provide some viscosity	Preserve-like, spreadable, thixotropic, (will not hold a cut surface)	Preserve-like, spreadable, thixotropic, (will not hold a cut surface)

With regards to texture, HM gels have a rigid, somewhat Jell-O-like texture, and will hold a cut surface. Above pH 3.5, HM pectin doesn't gel, and therefore provides some viscosity but no gel structure. LMC gels have a spreadable, preserve-like texture, with some increase in rigid gel structure as the pH is lowered below 3.4, and they generally will flow at a cut surface. LMA gels at pH values of 3.5 or higher have a very similar texture to LMC gels, with good spreadability. At pH values below 3.4, LMA gels are Jell-O-like or HM pectin-like, but somewhat more "rubbery".



The reason for the texture of an LM pectin gel being dependent on pH is shown in Figure 23. The pKa, or 50% ionization pH, of pectin is at pH 3.5. When the pH is below 3.5, there are a predominance of non-dissociated acid groups, which leads to more hydrogen bonding in the gel network. This gives rise to a more rigid, non-shear reversible gel network. When the pH is above 3.5, then there are a predominance of ionized acid groups, which leads to more calcium cross linking. This gives rise to a more spreadable, shear reversible gel network.

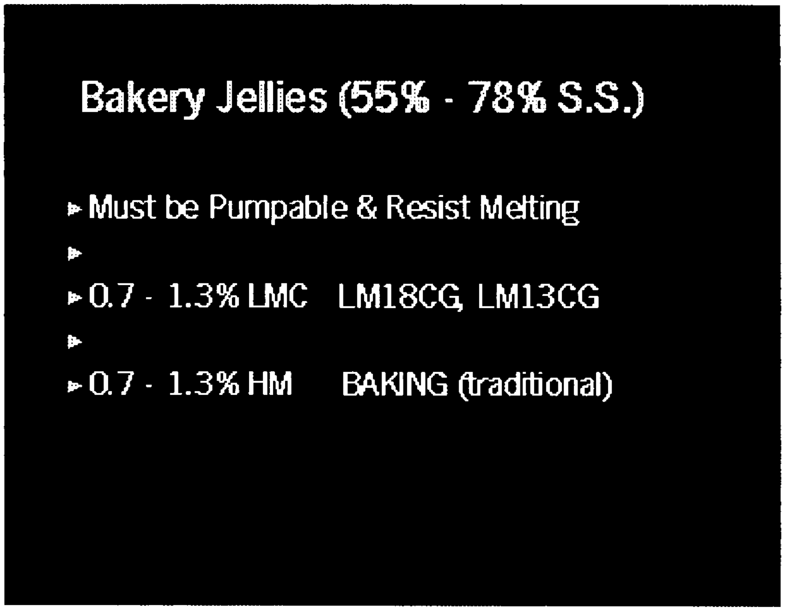
APPLICATIONS OF HM AND LM PECTIN

HM pectin is used for all traditional standard of identity jams, jellies, and preserves. These are made with a soluble solids of 65%, a final pH of 2.9 to 3.2, and generally contain 0.3% to 0.5% HM pectin. For the manufacturer who is filling strawberry preserves into one pound glass jars, the suspension of the fruit is critical. He fills the jars with 180F preserves to sterilize the inside of the jar, yet he doesn't want the strawberries floating up to the top half of the jar. Consumers think they are being cheated if the jar has fruit on the top half and what looks like jelly on the bottom half. To keep the fruit evenly suspended, the jam producer uses a rapid setting HM pectin. When the 180F product reaches the jar, it heats up the jar, and drops to 170F, and the rapid set pectin immediately begins to gel, thus keeping the fruit evenly suspended in the jar.

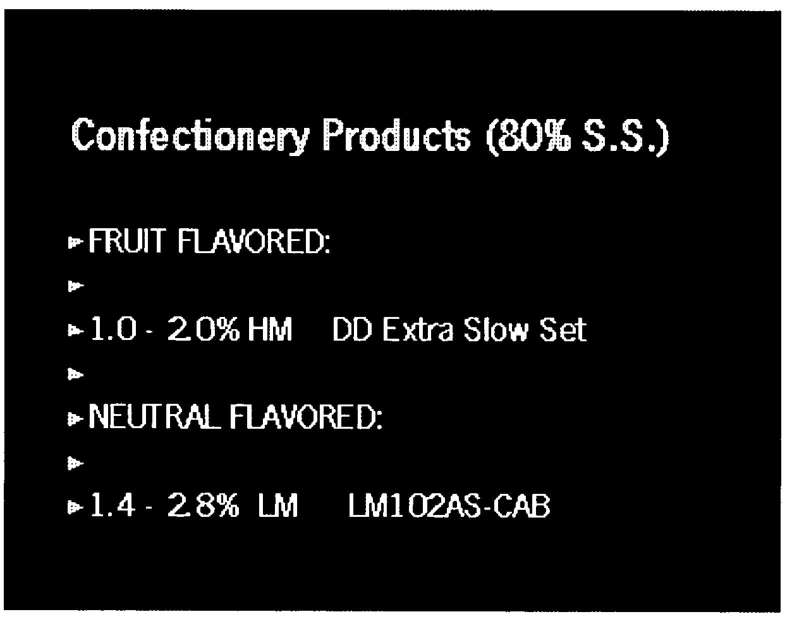
At the other end of the scale, the jelly producer is also filling his jars at 180F, but his filling machine tends to whip some air into the hot liquid jelly. Consumers don't like to see trapped air bubbles in jelly, as they think something is wrong with it (spoiled). The jelly producer therefore uses slow setting HM pectin. This allows at least fifteen minutes to go by after filling before the product begins to set, which is plenty of time for all the air bubbles to rise to the surface and break.

As soon as you move into the area of low or reduced calorie fruit spreads, you are outside of HM pectin gelling conditions, and you turn to LM pectin. The typical reduced calorie spread is 30% to 35% soluble solids, which represents half the calories of a standard of identity product. Also, there are "conserves", which are made only with

fruit and juice, and contain no refined sugar nor corn syrups. The conserves are generally in the 50% to 60% soluble solids range, which is just on the "ragged edge" of gelling conditions for HM pectin, which means that HM pectin will **not** be reproducible batch to batch. For the 30% soluble solids spreads, use a high calcium reactivity LM pectin, and for the conserves, use a medium reactivity LM pectin. As far as texture is concerned, I recommend a 50%/50% mixture of LMC and LMA, as this seems to give the best approximation to an HM pectin gel texture at these lower solids. We have even successfully made artificially sweetened jams and jellies at 6% to 10% solids with LM pectin, however I recommend that you add 0.1% to 0.4% locust bean gum to any formula below 25% soluble solids for long term syneresis control.



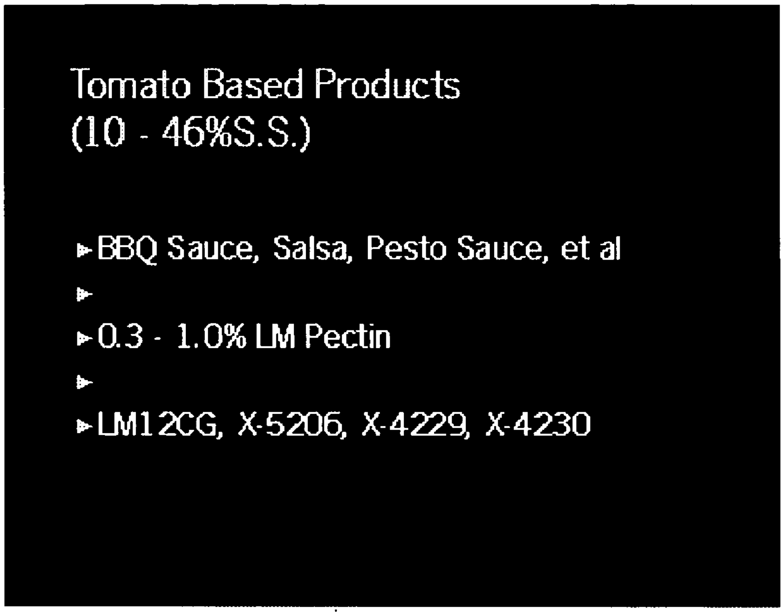
Traditional bakery jellies in the U.S. have been based on HM pectin, due to it's good thermal stability. These gels are prone to syneresis after being pumped or sheared, but this is only apparent at solids levels below 70%. If you want to make a pumpable (shear stable) heat stable bakery filling at soluble solids of 70% to as low as 50%, then you should consider formulating with LMC pectin.



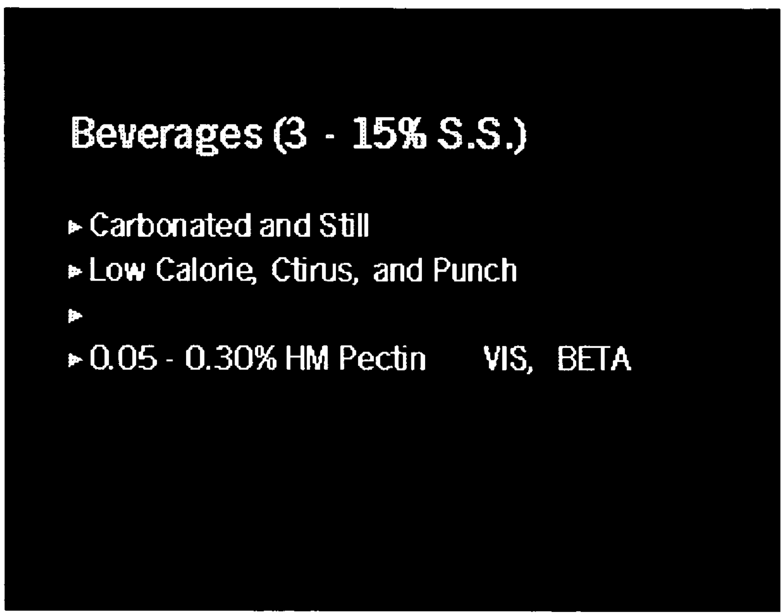
In the U.S., most candy is based on starch. In Europe, most candy is based on pectin. Pectin candy is a little more technically challenging to make, but it has the advantages of much better flavor release, and it can be cast directly into molds at 80% solids, cooled quickly, and demolded thirty minutes later. For a typical fruit flavored pectin candy, one would use an extra slow setting HM pectin, and would target on 80% final soluble solids with a final pH

of 3.5. This would give you 30 to 45 minutes at 180F to deposit the candy into molds.

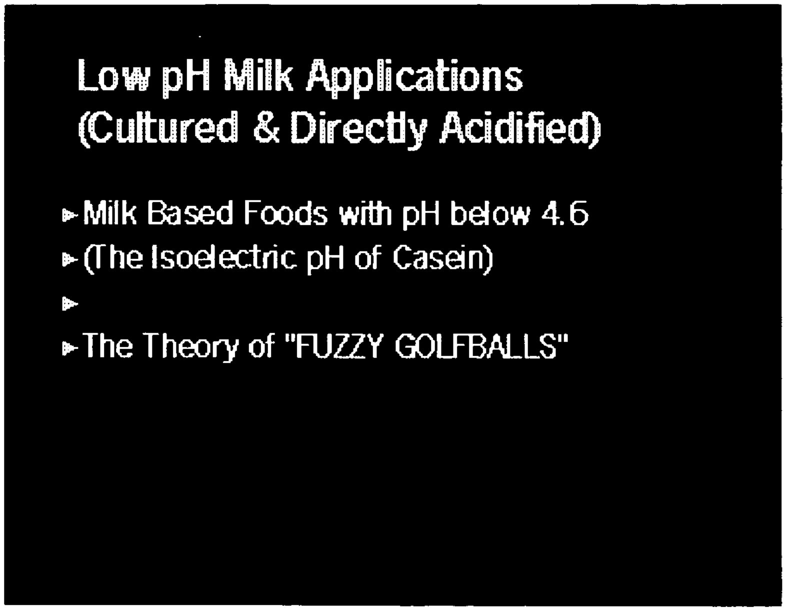
For the "neutral" flavors, such as vanilla, spearmint, licorice, etc, a buffered LM pectin would be appropriate. The buffer system is based on phosphate, and the final pH is around 4.2, so the final product does not taste sour, and is therefore compatible with neutral flavors.



Because of it's spreadable texture and it's excellent heat stability, LMC pectin can be used in tomato based products such as barbecue sauce and taco sauce, as an alternative to modified food starch.

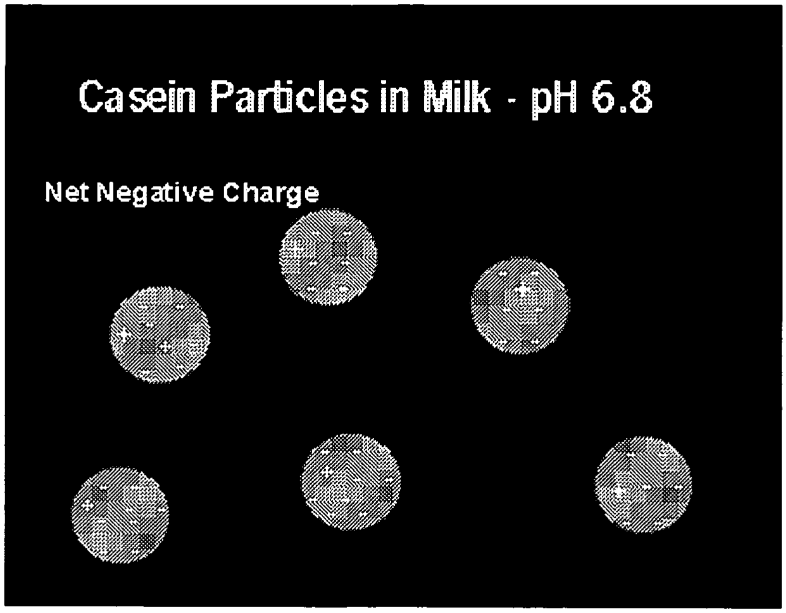


A typical beverage contains 10 to 15% dissolved sugar, and has a certain viscosity in the mouth as a result. When you make the diet version of the beverage, you take out the 15% sugar, and you put in 15% more water along with a tiny smidgen of artificial sweetener. The resulting beverage literally tastes as thin as water because it is water! A tenth of a percent HM pectin in a diet beverage can put back most of the texture you lost when you removed the sugar, without adding any significant calories. A dilute pectin solution will mimic the Newtonian behavior of a 15% sugar solution.

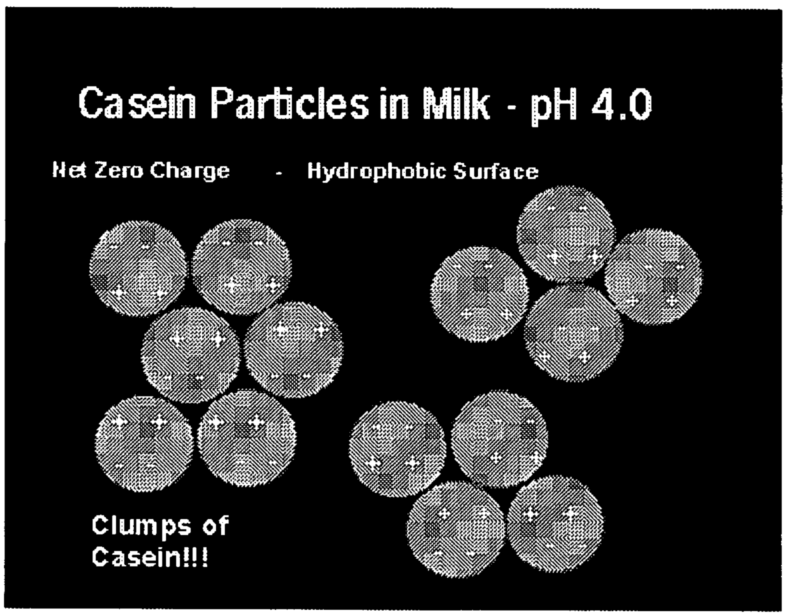


Pectin has another group of applications which are due to it's ability to stabilize protein which is being subjected to pH conditions below the protein's isoelectric pH. I will refer to this as the *"fuzzy golf ball"* theory.

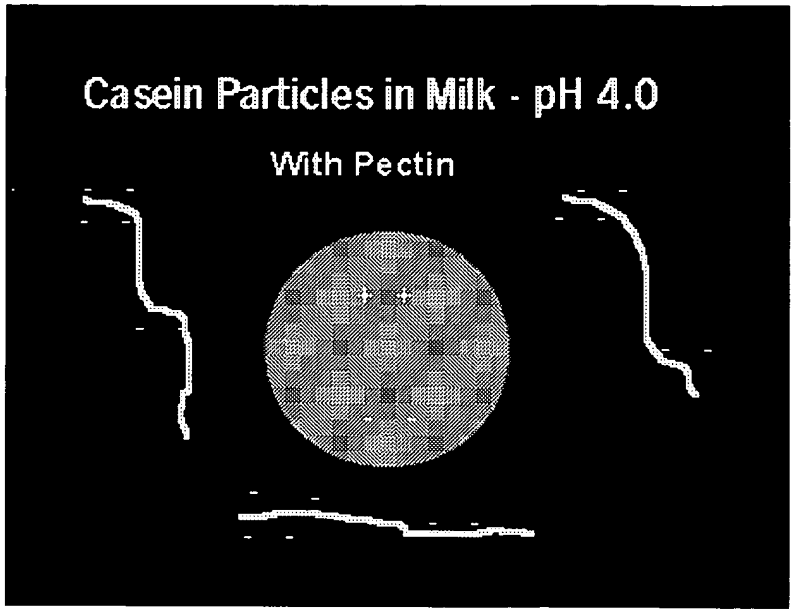
When you were little, did you ever pour your orange juice into your milk to see what would happen? The result is a rather nasty looking mixture, and the milk proteins quickly drop to the bottom of the glass. Now we have a way of making such a mixture and have it look appetizing, taste good, and be stable for months. Pectin can stabilize juice / milk and other directly acidified products, as well as cultured milk products such as yogurt, buttermilk, and sour cream. The theory goes like this:



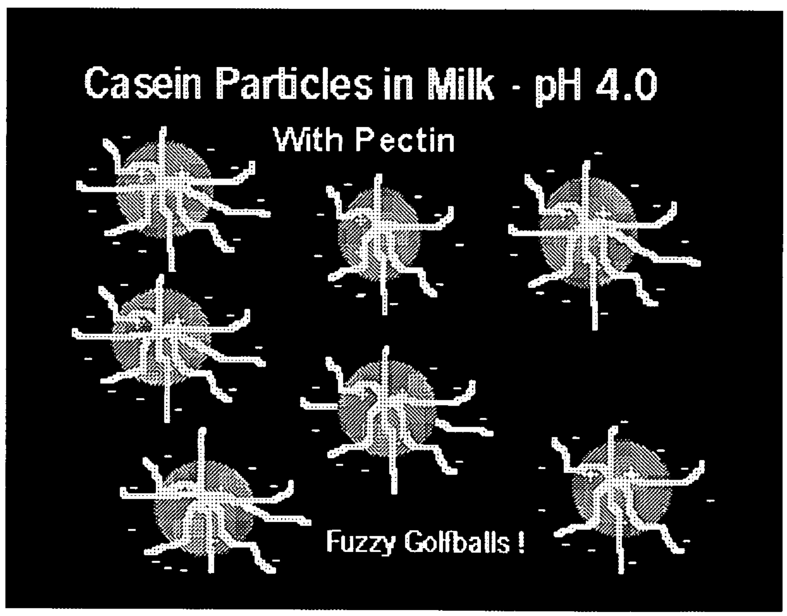
Milk is actually a suspension of water insoluble casein particles, which are very small in size. At milk's ambient pH of 6.8, the casein particles have a net negative charge, and they repel each other. The Brownian motion of the water molecules is sufficient to keep the casein particles in suspension indefinitely.

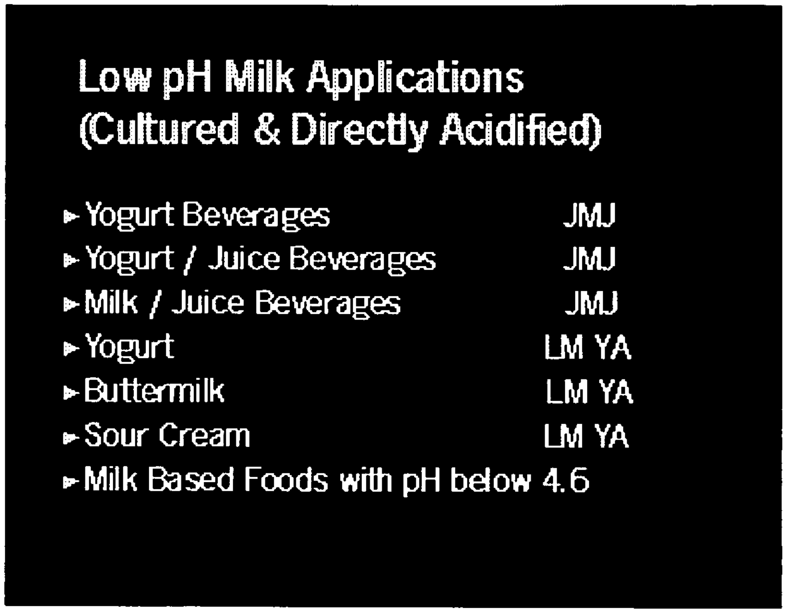


When the pH of milk is lowered by direct acidification or by the action of microorganisms on lactose, the casein particles lose their net negative charge. Although there are still positive and negative areas on the surface of each casein particle, the net charge on each particle becomes zero as the pH is lowered below about 4.6. When this happens, the casein particles begin to stick together in larger clumps, and Brownian motion will not keep these large clumps in suspension, so they settle to the bottom of the container.

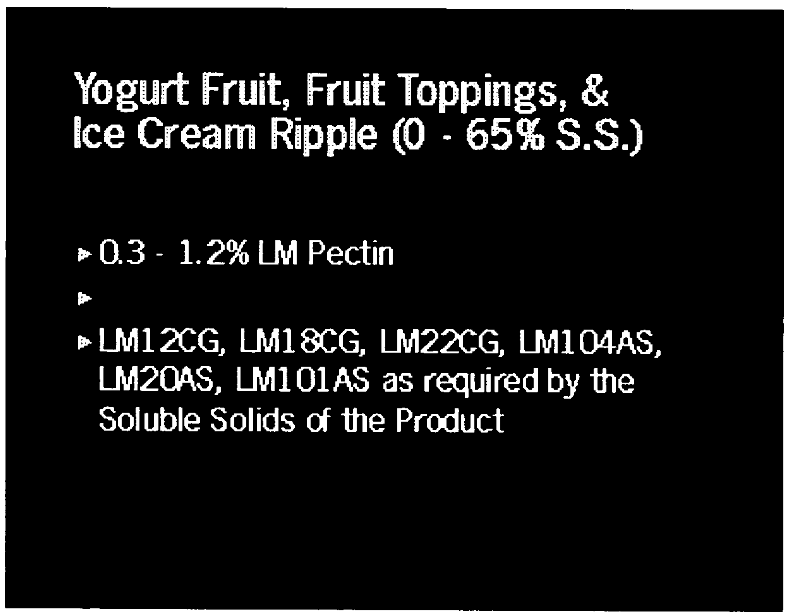


Pectin has a net negative charge at any pH above 3.5. In a low pH milk system, the negative pectin molecules will electrostatically stick to the positive areas on the casein particles, while avoiding the negative areas. If you visualize a casein particle as looking like a golf ball, then the pectin can be compared to short pieces of yarn. The electrostatic complex of casein and pectin ends up looking like a golf ball coated with yarn, hence the name ***"fuzzy golf ball"***.

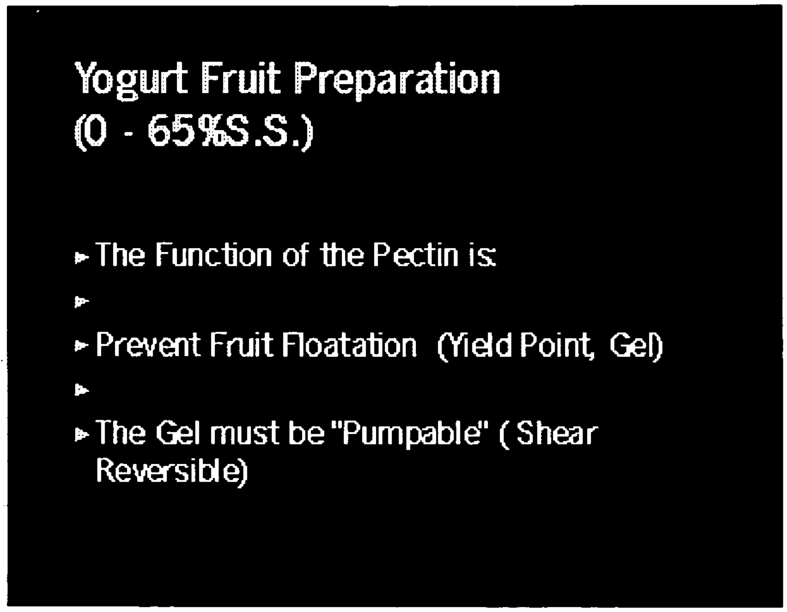




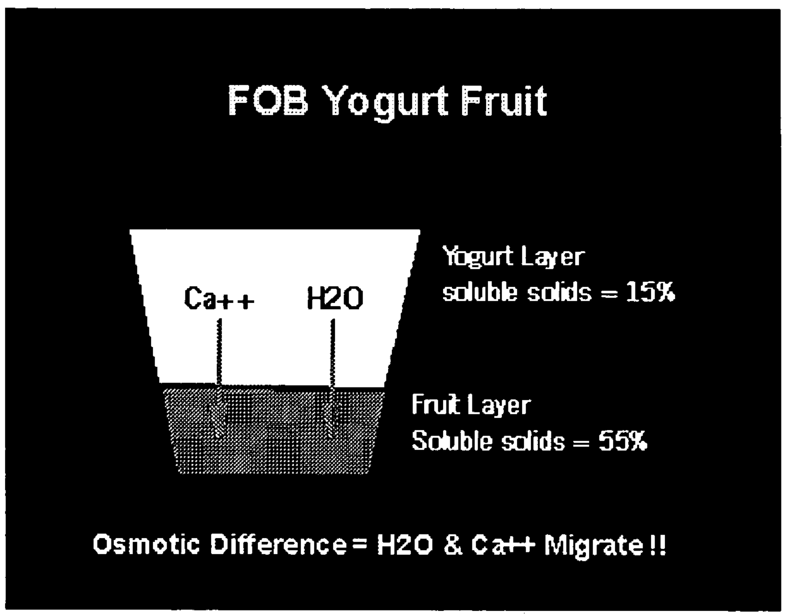
Pectin will also stabilize low pH soy milk as well as cow's milk. For directly acidified systems and for heat processed yogurt drinks, HM pectin works the best. For cultured products, LM pectin is more efficient.



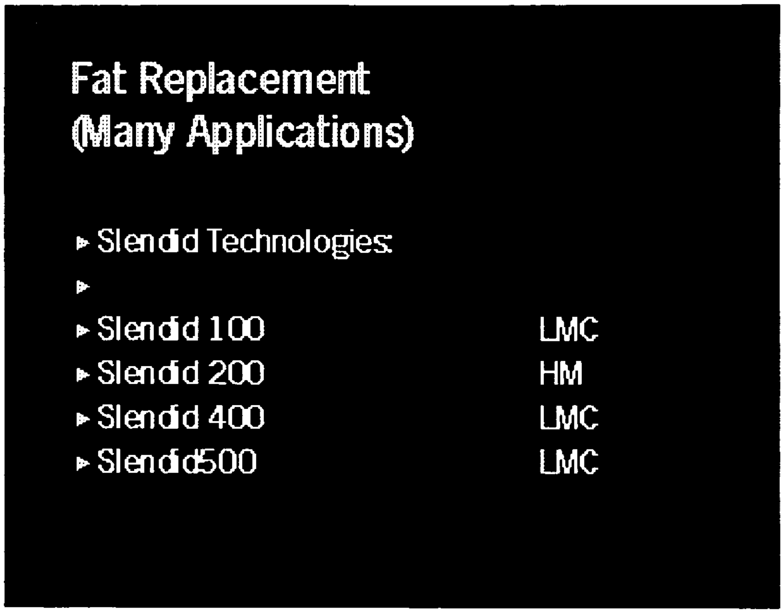
The last group of food applications are the yogurt fruit, ice cream toppings, variegate syrups, and related items. Of these, yogurt fruit is probably the most difficult technically.



Most yogurt fruit is packed into large stainless steel totes, and these totes typically hold one thousand pounds of fruit preparation or more. The fruit pieces must remain evenly suspended throughout the container, because the prep is pumped directly into the yogurt cups from the totes. In addition, there must be no tendency for syneresis to occur as a result of the shear that the fruit prep receives when it is pumped out of the tote and into the cups (i.e., it must be shear stable). LM pectin is perfectly suited for this application.



Fruit on the Bottom yogurt presents additional technical challenges. When the 15% soluble solids white layer is in contact with the ~55% soluble solids fruit layer, the osmotic difference causes water to migrate down into the fruit layer, taking with it all the ionic calcium present in the white layer (~1000 PPM). If the LM pectin in the fruit layer is not at calcium saturation when the fruit prep is made, then the LM pectin reaches saturation while it is under the yogurt, resulting in a very hard fruit layer which is difficult to stir into the yogurt. We refer to this hard fruit layer as a "hockey puck". To stop "hockey pucks" from occurring, one has to ensure that the LM pectin is calcium saturated at the time the fruit prep is made.



We have developed many Fat Replacer / Fat Holdout systems based on our pectin technology.

I hope that this "Introduction to Pectin" has been helpful and enlightening for you. If you have any technical or application questions regarding the use of pectin, please feel free to contact me:

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